Marina Chimica Acta, Oktober 2014
Jurusan Kimia FMIPA, Universitas

Vol. 15 No. 2
ISSN 1441-2132

AGE DETERMINATION OF LIMESTONE ROCKS LEANG - LEANG CAVE COMPILER THROUGH ACTIVITY MEASUREMENTS ^{14}C USING LSC

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ABSTRACT

Age Determination of Limestone of Leang - Leang Cave Through Activity Measurements of ^{14}C Using LSC. This research has been carried out by using limestone samples taken from Leang-Leang Cave, Maros. Limestone is one of the environmental samples with a constituent of calcite mineral ($CaCO_3$) which is derived from the remains of flora and fauna that has been weathered and petrified. Sample preparation was done physically and chemically. Preparation of chemically by using a mixture of NaOH with 30% H_2O_2 followed by a mixture of $HClO_4$ with 30% H_2O_2, and the last with HCl solution to produce a clean sample with a weight reduction of 3,36% - 4,44%. Carbonate matrix samples as CO_2 is produced by reaction with 85% H_3PO_4 and absorbed by 1M KOH solution as K_2CO_3. The total carbon in the sample solution is 0,6144 - 0,9696 grams obtained through titration method. Radiocarbon dating method based on the measurement of the specific activity of the samples obtained from the results of counts LSC (*Liquid Scintilation Counter)* Hidex 300 SL. The specific activity of both of samples were 1,609±0,0359 DPM/g C and 7,718±0,109 DPM/g C. Age of both of limestone samples which were calculated from the specific activity were 25.607,403 ± 919,305 years and 8457,792 ± 921,899 years for BG I and BG II, respectively.

Keywords: Limestone, LSC (*Liquid Scintilation Counting*), radiocarbon dating, specific activity.

INTRODUCTION

Indonesia is an archipelago located in a very strategic position in the equatorial region that connects the continents of Australia and Asia and the Pacific and Indian oceans. The climate is pleasant, varied natural wealth that make Indonesia as a country which has great potential in natural resources (SDA) to develop into a powerful country in the Asia - Pacific (Suhadi et al, 1998). One of the natural wealth of Indonesia located in South Sulawesi, especially those in areas such as Maros - Pangkep the karst rock stood firm. Maros karst region - Pangkep upper part of some major rivers, including the River upstream Pangkep Pute River and River Bantimurung/Maros (Prawitosari, 2011). According to Ahmad (2001) in Daryanto and Oktariadi (2009) Maros karst region is

part of a mountain range that stretches from Maros area to the north to the entrance to the area Pangkep. This area formed from the dissolution of limestone outside as the hills towering upright, and which occurs in the phenomenon which forms a very beautiful and unique, such as the formation of caves horizontal and vertical caves, stalactite, stalagmite, and others. Growing limestone is characterized by a more solid and resistant to denudation processes and form steep hills and higher elevations outside the karst formations in the form of a hill.

Organic limestone is a collection of residual flora and fauna that have died (fossil) and sedimented. In his lifetime flora and fauna require elements Ca, Mg, O and C are present in the water. Chemical processes that occur on the flora and fauna after death into a fossil and mineral crystals forming system without changing the physical form of fossils. In mineralogy of the fossil is composed of the mineral calcite ($CaCO_3$) and dolomite [$CaMg(CO_3)_2$]. In general, the fossil fauna containing calcite magnesium grading (4% -16%)mol $MgCO_3$, while the fossil flora of approximately (7,7% -28,75%)mol $MgCO_3$. Mineral calcite or dolomite mostly small (± 0,2 microns or more) is called carbonate mud (Harjanto, 2001). Because of the small size, the optical and physical properties relaif same, so it is difficult to distinguish the two minerals. Through the collection of fossils geological processes into limestone (Hiskia and Tupamahu, 2001).

Carbon content contained in these rocks be the deciding factor in determining the age of a sample. Carbon is what is referred to as radiocarbon. Radiocarbon withdrawal by Faure (1986) in Siregar (2011) is the radiometric method that can be used to determine the absolute age of a sample by age 50.000 years ago. Method of age determination can only be made on materials that contain the element carbon. Elemental carbon isotope ^{14}C used is contained in atmospheric CO_2 bound in a compound. ^{14}C isotope is produced by the reaction of cosmic rays with nitrogen.

$$^{1}_{0}n + {}^{14}_{7}N \rightarrow {}^{14}_{6}C + {}^{1}_{1}p$$

Isotope is called radiocarbon. Carbon isotope widely distributed in nature and found in every organic compound.

One method that is very popular in determination age of an object is a method of LSC (*Liquid Scintillation Counting*). This method works trace radioisotopes, in particular β emitting isotopes on the basis of the interaction of a solution of organic compounds that can interact with radiation (Salam, 1993).

The principle of the liquid scintillation counting method, samples containing radionuclides dissolved or suspended in a solution of scintillator (cocktail) that fits inside the glass or plastic vial. Radioactive particles in the sample is dissolved in a scintillator solution will collide with molecules of the solvent causes solvent molecules become excited and led to the scintillator molecules emit photons. The photons then detected by the PMT, so that the resulting electrical pulses are proportional to the energy of radioactive particles (Tjahaja and Mutiah, 2000).

Liquid scintillation counting method is usually only used for the enumeration of β low -energy radiation, such as ^{3}H and ^{14}C, but with the development of a liquid scintillation counter instrument, this method can be used also for the enumeration of total α and β. Advantages compared with the previous method is a liquid dosage form so as to facilitate sample homogeneously soluble so there is no effect of self-absorption and more efficient sample detection time (Tjahaja and Mutiah, 2000)

METHODS

Time and Location

This study conducted in October-December 2013 in the Radiation Chemistry Laboratory, Department of Chemistry, Faculty

of Mathematics and Natural Sciences, University of Hasanuddin, Makassar.

Tools and Materials

Hammers, files, LSC (Liquid Scintillation Counter) Hidex 300 SL and limestone samples from Leang-Leang Cave.

Methods
Sampling Limestone Rock

Limestone samples were taken using a file and a hammer at two point coordinates. Then, pieces of limestone rocks inserted into the plastic to be brought into the laboratory.

Washing Limestone Rock Samples

Limestone samples washing done in two stages, such as:

1. Physical wash

Limestone samples cleaned with water and brushed and followed by rinse. After that, placed in the cup and allowed to stand a few days for the drying process. Limestone that has been dried and then broken into small pieces and weighing is done first to determine the initial weight.

2. Chemical leaching

Washing step is then performed by immersing samples of limestone that had been weighed in a mixed 50/50 30 % H_2O_2 and 1 N NaOH in a 100 mL beaker is placed in an ultrasonic for ±10 minutes and proceed with the separation of wash solution, then rinsed with distilled water several times until the rest of the wash solution is lost. The next stage, limestone samples immersed back into the mix 50/50 30 % H_2O_2 and 1 N $HClO_4$ in a 100 mL beaker for ±30 seconds, and then separated again from the wash solution. The last stage of the washing process is by soaking the samples in 6 N HCl solution for 15-60 seconds and separated again from the wash solution, then rinsed again limestone samples with distilled water several times. Furthermore, limestone is dried in an oven at a temperature of 60 °C until the dry rocks. After that, weighed again to determine the weight % limestone lost during the washing process takes place.

Analysis of CO_2

Each rock sample that has been dried and finely crushed to 10 grams were taken for analysis of ^{14}C or carbon content of the total. 10 gram samples that have been smoothed placed in a round bottom flask, then rinsed thoroughly with a solution of 85 % H_3PO_4 were placed in a separating funnel until all the powder samples completely reacted with H_3PO_4. Then, the resulting CO_2 gas is passed into a solution of 10 % KOH, as shown in Figure 1.

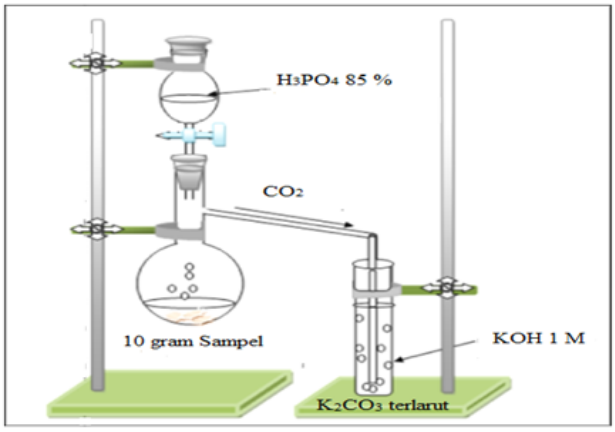


Figure 1. Design tools as CO_2 separation carbonate limestone sample.

Determination of Total Carbon

Solution of K_2CO_3 dissolved pipetted 10 mL into erlenmeyer for titration with 5 M

HCl solution after the addition of a few drops of indicator MO until the color changes from brown to red. Subsequently, 10 mL of solution pipetted into beaker K_2CO_3 for 10 % $BaCl_2$ solution is added to precipitate (*saturated*). Subsequently, the precipitate and filtrate are separated by means of filtered, and the filtrate was pipetted into a 10 mL erlenmeyer to titrated with 5 M HCl solution with the addition of a few drops of indicator PP until the color changes from purple to clear.

$$t = \frac{t_{1/2}}{ln\,2} ln \frac{A_0}{A}$$

Enumeration Limestone Rock Samples

Enumeration of the samples was done by 8 mL of sample solution is pipetted into a glass vial of 20 mL, then added 12 mL of scintillator and shaken until homogeneous. Furthermore, the sample with the LSC chopped Hidex 300 SL made with interval time 1-300 minutes.

Calculation of Absolute Age

Age calculation is done using the formula:

A = Radioactivity of ^{14}C isotopes in the sample

A_o = Radioactivity isotope ^{14}C during the life of plants or animals 15,3 DPM (Libby, 1960)

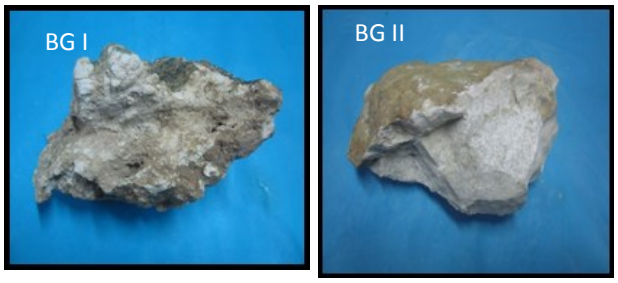
$t_{1/2}$ = Half-life of ^{14}C 5730 years

ln2 = 0,693

RESULTS AND DISCUSSION

Sampling Location

Sampling sites located in the limestone Leang-Leang cave Maros, South Sulawesi Province. This location is one of the protected historic attractions and are within the scope of Maros, especially at Leang - Leang village. Sampling is done on two limestone certain point. The selection of this location as a place of sampling because the limestone Leang-Leang Cave is one of the heritage assets within 20 km of the town of Maros or 44 Km from the city of Makassar and can be reached for ± 1 hour drive. There are two limestone samples taken from the study site. Both samples of limestone



can be seen in Figure 2 below.

Limestone sampling technique done in two ways, namely taking rocks that have been separated from the cave walls and mossy limestone rocks for samples BG I to coordinate S: 04°39'37,4" and E: 119°27'14,6", while the limestone samples BG II to coordinate S: 04°39'41,8" and E: 119°27'21,9" taken using a file and a hammer on the walls of the cave are still strongly attached.

Both the limestone samples will be determined the contribution of ^{14}C activity contained there in are subsequently converted into a part-time equation for calculating the length of the sample had died. This was done to compare the age of two limestone samples which assumed that the proportion of the isotope ^{14}C in the air is relatively constant before contributing to the tissue of living things. At the time of living creatures has died, the equilibrium will be stopped and the activity in the tissues of living beings who have died as a result of

Fig. 2. Limestone samples BG I and BG II pri-

or to destruction and leaching

the decay of radioactivity will decrease contin uously.

Cleaning Sample

Pretreatment samples of limestone are physically laundering. Washing is done in stages beginning with the washing with water followed by washing with distilled water is used to remove impurities that are easily lost as soil. The next stage is a chemical leaching is by immersing samples of limestone into a solution of 30% H_2O_2 and 1 N NaOH (50/50) in a 100 mL beaker was placed in an ultrasonic apparatus for 10 minutes to speed up the washing process by giving vibrations the base and walls of the sample container during the washing process takes place. Meanwhile, wash with a solution of 30 % H_2O_2 and $HClO_4$ 1 % (50/50) in a 100 mL beaker for ± 30 seconds intended to oxidize other pollutant sources are not lost during washing with 1 N NaOH solution and eliminate the dyes contained limestone rock samples to be pure white. Last laundering using chemicals is with 6 N HCl which serves to eliminate impurities and secondary carbon sources from the sample surface which has not been lost on the previous washing. Washing with 6 N HCl is done during 30-60 seconds. The missing part of the sample is a natural contamination accumulated over limestone in contact with air and surrounding environment. Results of treatment of the samples is shown in Table 3.

Table 3. The data resulting from leaching limestone samples

Samples	Coordinate Point		Washing Weight	
	S	E	Physics (g)	Chemistry (g)
BG I	04°39'37,4"	119°27'14,6"	87,956	85,000
BG II	04°39'41,8"	119°27'21,9"	80,176	76,617

Weighing the two limestone samples that BG I and BG II is done after the sample was dried. Based on data from the weighing results as shown in Table 3 above, the difference in initial weight gained weight after weighing the sample for each sample limestone BG I 2,956 grams, while the limestone samples BG II 3,559 grams. The weight difference in the two samples is the number of sample weight lost during the washing is done , which is about 3,36 % limestone samples BG I and 4,44 % for limestone samples BG II of weight initially. The results of this washing produce sample weight lost is not much different from the one described by Adkinds et al, (2002) that the process of washing the sample with chemical compounds such as the above can eliminate the weight of the samples ranged from 5-10 % by weight before. That is limestone samples have small amounts of impurity components when compared to other environmental samples that have a number of small pores on its surface.

Analysis of CO_2

Chemical components into the main constituent in limestone is $CaCO_3$. Carbonate contained in the limestone can be separated by treatment with 85 % H_3PO_4. This step can be done after the sample has been crushed limestone to be smooth, so that the contact area between H_3PO_4 85 % with the sample becomes

more widespread and the reaction can take place quickly. The reaction that occurs when a sample of limestone was added with 85 % H_3PO_4 slowly will produce gas bubbles on the surface of the sample, as shown in the reaction below.

$$CaCO_{3(s)}+H_3PO_{4(l)} \rightarrow CaHPO_{4(s)} +CO_2(g)+ H_2O_{(l)}$$

The gas bubbles are released and the CO_2 gas is passed into a solution of 1 M KOH absorbent. In this process, not all the CO_2 gas is passed into a solution of 1 M KOH absorber capable arrested. This can be seen from the presence of CO_2 gas bubbles through the surface of the absorbent solution. CO_2 is absorbed by the absorbent solution of K_2CO_3 dissolved KOH will produce.

$$CO_{2(g)} + KOH_{(l)} \rightarrow K_2CO_{3(l)} + H_2O_{(l)}$$

K_2CO_3 solution is a solution of the sample to be measured by LSC (*Liquid Scintilation Counter*) to determine the amount of ^{14}C activity.

Determination of Total Carbon

Determination of total carbon contained in the limestone samples can be done by titration. This method is very supportive in this study. The total mass of the carbon samples obtained from BG I as much as 0,6144 grams, whereas for sample BG II as 0,9696 grams. According Djuhariningrum and Rusmadi (2004) that the carbon content in weight of $CaCO_3$ that there is 12% carbon by weight. Calculation of the total carbon in limestone samples need to be done to get the value of the specific activity of ^{14}C contained in the sample. The specific activity of ^{14}C itself is expressed in units of disintegrations per unit mass of carbon (DPM/gC).

Sample Enumeration

The specialty tools Hidex LSC 300 SL when compared to other tools is a device enumerator MikroWin 2000 software system that is able to provide results of its absolute chopped. Enumerators sample solution is performed using a set of tools LSC (*Liquid Scintilation Counter*) Hidex 300 SL has an important role in the detection of β particles emitted from the radioisotope ^{14}C originating from the sample. Sample enumeration results with these tools will get the amount of ^{14}C activity is expressed in units *Disintigrations per Minute* (DPM) and the results obtained during enumeration is done in units of counts *Counts per Minute* (CPM). This enumeration method works trace radioisotopes, in particular β emitting isotopes on the basis of the interaction of a solution of organic compounds that can interact with radiation (Salam, 1993).

Enumeration can be done after the addition of 12 mL of scintillator solution in 8 mL of sample solution in a 20 mL vial. This process is carried out in poorly lit room to avoid contamination of free air containing CO_2. Then, enumerated in the span of 1-300 minutes. Scintillator solution used contains two components, namely primary and secondary solvent. Radioactive particles contained in the sample is dissolved in a scintillator solution will collide with molecules of the solvent so that the solvent molecules become excited and emit photons. The photons then detected by the PMT, so that the resulting electrical pulses are proportional to the energy of radioactive particles. According Satrio (2009) This method is done by counting the CO_2 absorbed by the absorbent solution, so that the activity of samples containing ^{14}C in CO_2 directly enumerated. Analysis of samples by this method involves the scintillator solution that will collide with the solvent molecules to excited. At this point the energy is released in the form of photons or

light flicker. The flicker of light has a specific wavelength and when it comes to fotokatode layers in PMT (*Photo Multiplier Tube)* will release electrons from the layer. This will be multiplied by the electron dinode dinode contained in the PMT and eventually these electrons will be collected on anode in the form of electrical pulses. Data results of the second sample enumeration limestone can be seen in Table 2 below.

Table 2. The data sample enumeration limestone BG I and BG II with a span of 1-300 minutes

No	Times (Minutes)	BG I Activity		BG II Activity	
		CPMs	TDCRs	CPMs	TDCRs
1.	1	224,520	0,824	334,640	0,882
2.	15	221,820	0,740	327,180	0,772
3.	30	215,850	0,816	319,040	0,878
4.	60	217,020	0,853	317,840	0,863
5.	120	218,600	0,779	322,030	0,732
6.	180	221,550	0,724	323,030	0,726
7.	240	220,010	0,731	319,430	0,697
8.	300	219,769	0,853	319,030	0,701
Average		219,892	0,790	322,778	0,781

Based on Table 2 above shows the results of counts on which the shredded three minutes for 30 minutes for the first BG sample count values decreased from the previous chopped, shredded and then further increased. Increasing the results of counts due to the effect of the instability of the phase between the sample solution with a solution of K_2CO_3 dissolved scintillator is not homogeneous, so the results of counts that fluctuate between 215,850 to 224,520 CPM with an average of 219,892 chopped CPM. The results of counts if made in the shape of the graph will look like in Figure 3.

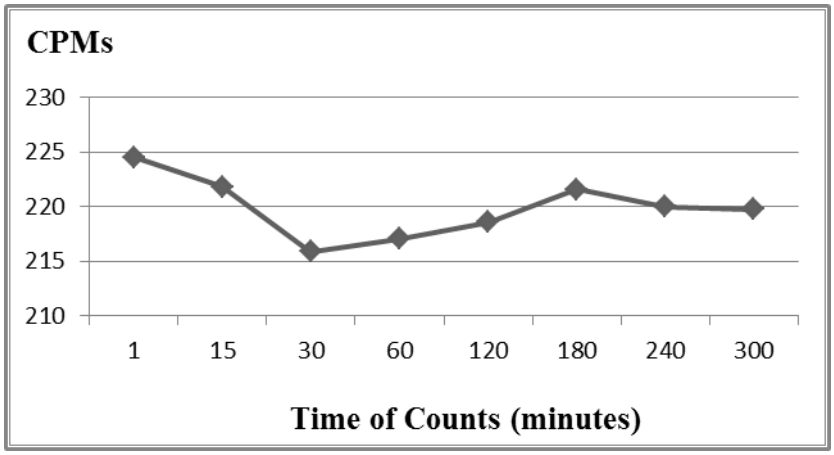


Figure 3.

Graph showing the relationship of CPM results limestone samples BG I against time

Likewise with BG II samples have initial count values are very high and decreased in the fourth when chopped for 60 minutes. Similarly, the sample BG I, BG II sample enumeration results also fluctuated between 317,840 to 334,640 CPM with average chopped 322,778 CPM. The results of counts if made in the shape of the graph will look like in Figure 4.

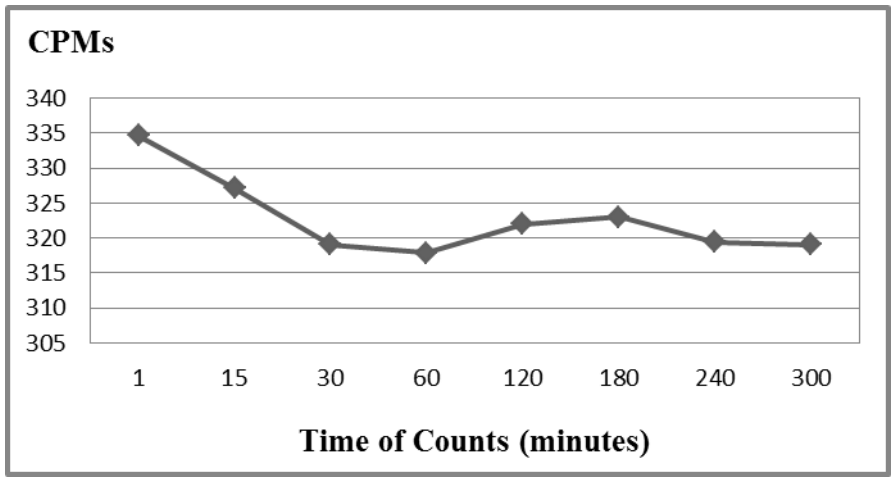


Figure 4. Graph showing the relationship of CPM results limestone samples BG II against time chopped

Although both samples above show the results with values berfluaktuasi chopped or rising slowly, it does not mean cacahannya value will remain constant over the average value of counts. This can be seen in the re-enumeration of each sample along with chopped blank BG contained in Table 3 and Table 4.

No	Times (minutes)	BG I Activity		Blank Activity
		CPM	TDCR	CPM
1	30	214,310	0,827	213,810
2		218,280	0,844	216,020
3		215,210	0,822	215,120
4		219,250	0,848	217,420
5		215,650	0,841	215,610
6		217,420	0,849	216,210
7		217,250	0,852	217,210
8		214,680	0,852	214,110
9		219,820	0,857	219,720
10		219,950	0,857	218,210
Average		217,182	0,846	216,344

Table 3.
The data enumeration BG I and blank samples for 30 minutes 10 times repetition

Based on Table 3 above, the data looks chopped repetition which gives an average yield of counts 217,182 CPM with enumeration efficiency of 84,6% for the BG I sample. The efficiency of sample enumeration is consistent with that described by Zhu and Yang (1995) that the efficiency of counts for β energy range 80-85 %.

While the results of counts blank that serves as a correction factor to the results of chopped samples gave an average yield of 216,344 CPM chopped smaller than chopped sample results. This means that the digital samples with a blank has an average increment of counts of 0,838 CPM.

Table 4. The data sample enumeration BG II for 60 minutes 10 times repetition

No	Times (minutes)	BG II Activity		Blank Activity
		CPM	TDCR	CPM
1.	60	314,640	0,888	304,430
2.		298,040	0,879	297,520
3.		301,740	0,883	297,570
4.		309,140	0,887	298,760
5.		305,840	0,901	299,220
6.		308,740	0,876	298,290
7.		314,700	0,908	311,830
8		311,740	0,880	299,960
9		308,430	0,828	300,960
10		313,760	0,722	312,690
Average		308,677	0,865	302,123

While the limestone samples for enumeration BG II as shown in Table 4 above, the results of chopped has variation although long used the same time. Counts range between 298,040-314,700 CPM which has an average of counts per minute 308,677 CPM with approximately 86,5 % counting efficiency. It is not much different from that described by Zhu and Yang (1995) that the enumeration for energy efficiency β ranged from 80-85% .

This can be explained by assuming that the decay is a statistical event. This suggests that the statistical properties of the atoms that will decay in the next second can not be ascertained because only a probability (Syahrir, 2001). Counting efficiency may be reduced because of the symptoms of burnout (*quenching*) which occurs due to the presence of oxygen or impurities in the solvent container scintillation vials. Blackout effect on β spectrum is the fluorescence spectrum shifts toward lower energy. The larger outage occurs, the fluorescence spectrum increasingly shifted to the left or toward the lower energy (Yarianto et al , 2001).

The use of blank enumeration herein is intended to determine the contribution of radiation from liquid scintillation counting environments that are not sampled. Results of counts blank is also used as a correction factor to the digital samples.

Determination of Specific Activity Sample

The specific activity of limestone samples can be determined from the difference between the results of counts *Counts Per Minute* (CPM) against the results of counts of blank samples generated as a correction factor to the outcome of chopped samples divided by the efficiency of enumeration *Triple Double To Coincidance Ratio* (TDCR) were converted into units of *Disintegration per Minute* (DPM), then divided by the total carbon contained in 8 mL of sample was mixed with 12 mL of scintillator. The specific activity of the sample is the result of the CPM samples were corrected by blank. Thus, from the results of the exposure calculation technique as described above, the specific activity of the sample can be determined limestone. The specific activity of two limestone samples that BG I and BG II is expressed in units of DPM/gC, as found in Table 5.

Table 5. The data on average specific activity of ^{14}C samples limestone BG I and BG II

Samples	DPM	C-total (g)	As (DPM/gC)
BG I	0,989	0,6144	1,609 ± 0,0359
BG II	7,484	0,9696	7,718 ± 0,109

Based on data enumeration sample results in Table 5 above shows the specific activity of ^{14}C in samples of limestone BG I is 1,609 ± 0,0359 DPM/gC, and samples of limestone BG II 7,718 ± 0,109 DPM/gC. Activity obtained indicates the magnitude of the decay of carbon atoms that take place every minute (DPM) per gram of carbon. Both the specific activity data obtained samples showed a very large difference between the two and is lower than the value of the average specific activity of modern living plants as the result of research conducted by Libby (1960) is often used as an early activity in the determination of the age of the sample environment ie 15,3 DPM/gC.

Age Determination of Limestone Rock Samples

Age of limestone samples can be determined by converting the measurement results obtained by the specific activity of each sample limestone into equation calculation of absolute age above.

From equation above obtained the second age limestone Leang-Leang cave constituent, as found in Table 6.

Table 6. The data on the calculation of age limestone through the measurement of ^{14}C activity

Samples	Age (years)
Limestone (BG I)	25.607,403 ± 919,305
Limestone (BG II)	8457,792 ± 921,899

Calculation of ^{14}C activity in the determination of the age of two limestone samples by liquid scintillation method gives results for each sample age limestone BG I 25.607,403 ± 919,305 years, while the limestone samples BG II 8457,792 ± 921,899 years. As has been discussed earlier that BG I limestone samples are samples that have been detached from the wall of the cave and have mossy physically older than BG II the limestone samples were still strongly attached to the wall of the cave and the relatively younger, so the way from using hammer and miserly. Both age limestone samples were counted is the age of the sample length is dead or no longer live shows activity that counted before 2013. As for knowing when the sample is dead , then the age of death decreased by the length of the sample calendar year since it began in the 2013.

CONCLUSION

Age of limestone samples BG I calculated based on data specific activity of ^{14}C is 25607,403 ± 919,305 years, while the age of the rock sample limestone BG II is younger, which is 8457,792 ± 921,899 years.

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